7. ANALYTICAL METHODS

The purpose of this chapter is to describe the analytical methods that are available for detecting, measuring, and/or monitoring polybrominated biphenyls and polybrominated diphenyls ethers, their metabolites, and other biomarkers of exposure and effect to polybrominated biphenyls and polybrominated diphenyls ethers. The intent is not to provide an exhaustive list of analytical methods. Rather, the intention is to identify well-established methods that are used as the standard methods of analysis. Many of the analytical methods used for environmental samples are the methods approved by federal agencies and organizations such as EPA and the National Institute for Occupational Safety and Health (NIOSH). Other methods presented in this chapter are those that are approved by groups such as the Association of Official Analytical Chemists (AOAC) and the American Public Health Association (APHA). Additionally, analytical methods are included that modify previously used methods to obtain lower detection limits and/or to improve accuracy and precision.

Polybrominated biphenyl (PBBs) and polybrominated diphenyl ether (PBDEs) are analyzed in environmental and biological samples by methods quite similar to those used for polychlorinated biphenyls (PCBs) (de Kok et al. 1977; Fries 1985b; Pomerantz et al. 1978). The analysis methodology includes several steps: sample collection and storage, extraction, cleanup, and determination. Care must be taken to assure that the sample collection follows quality assurance protocols and that equipment and containers are free from contamination. Most sample collections are by grab sampling; however, PBBs and PBDEs may be concentrated from water onto sorbents. PBBs and PBDEs are typically separated from the biological and environmental media by extraction with organic solvents. Cleanup steps are necessary to remove compounds that may interfere with the determination. Lipids (e.g., oils and fats) are removed with concentrated sulfuric acids. Chromatography (e.g., gel permeation, silica gel, Florisil) is used to remove other matrix interferences and to fractionate samples. The identification and quantitation of PBBs and PBDEs are most often accomplished by gas chromatographic (GC) techniques. Capillary or high resolution gas chromatography (HRGC) columns capable of separating a substantial proportion of the congeners are indispensable, and GC detectors possessing high selectivity and sensitivity for the PBBs and PBDEs are required. The more universal and less sensitive flame-ionization detector (FID) is used much less often than the electron capture detector (ECD), which has exceptional sensitivity to multiply brominated compounds. The mass spectrometer (MS) detectors have sensitivities somewhat lower than ECD, and they have even greater selectivity for PBBs and PBDEs and can distinguish and individually measure homologs that may coelute on a particular HRGC column. A recent method of detection is electron capture negative ionization (ECNI) as an ionization technique in combination with GC-MS analysis (de Boer et al 2000). This method is advantageous because it offers a high sensitivity for

compounds with four or more bromine atoms. The sensitivity is approximately 10 times higher than with the use of the ECD. The small number of certified reference standards that are available for individual PBB and PBDE congeners poses a problem for analysis, since comparative analysis is often based on technical mixture equivalents rather than by comparison of individual congeners.

de Kok et al. (1979) described several analysis methods for analysis of commercial PBDEs such as GC-MS, high-performance liquid chromatography (HPLC), reversed-phase thin layer chromatography, and ultraviolet (UV) spectrometry.

7.1 BIOLOGICAL MATERIALS

Methods for the determination of organobromine compounds such as PBBs and PBDEs generally consist of the following steps: extraction of the analyte from the sample matrix; clean-up to remove interfering compounds; and analysis (separation and quantitation). The primary method of analysis is GC coupled with ECD or MS. Analytical methods have been developed for the determination of PBBs and PBDEs in blood or serum, urine, feces, adipose tissue, liver, and breast milk. The methods for determining PBB and PBDE residues in biological samples are given in Tables 7-1 and 7-2, respectively.

Polybrominated Biphenyls. Residues in biological samples can be extracted using hexane/ether, petroleum ether/diethyl ether, toluene/ethyl acetate, or methylene chloride (Burse et al. 1980; Domino et al. 1980; Fawkes et al. 1982; Fehringer 1975b; Wolff et al. 1979b). Elution of samples on a florisil column, which is used for the cleanup of extracts with petroleum ether, separates PBBs from interfering substances (Pomerantz et al. 1978). As in the case of PCBs, the solvent(s) used for the extraction of a sample and the method used for the cleanup of an extract is dependent on the sample matrix (Pomerantz et al. 1978). Quantitation is usually done by GC. The major difference between the methods for the determination of PCBs and PBBs arises from the lower volatility of PBBs compared to PCBs. Due to the lower volatility of PBBs, the GC method is performed at a higher temperature and low liquid phase load of the stationary phase. Capillary columns are required for the separation of the individual congeners in a mixture (Robertson et al. 1983b). However, decabromobiphenyl is so nonvolatile that a very short capillary column and high carrier gas linear velocity are required, which reduces the advantage of the capillary column over the packed column (Farrell 1980). Peaks from individual congeners of PBBs are detected and quantified with the ECD (Robertson et al. 1983b). In general, retention time in gas chromatographic columns and response of ECD increase with increasing bromination. PBB residues in a sample can be confirmed by thin layer chromatography, photochemical alteration method, halogen-

Table 7-1. Analytical Methods for Determining PBBs in Biological Materials

Sample matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Serum	Extract denatured sample with hexane-ethyl ether; clean up by Florisil column chromatography	GC-ECD	1 µg/L	100.6–106.8 at 100 µg/L	Burse et al. 1980
Serum	Extract denatured sample with hexane-ether; clean up by Florisil column chromatography	GC-ECD	1 µg/kg	86–92	Wolff et al. 1979
Plasma	Extract denatured sample with petroleum ether-ethyl ether; clean up by Florisil and silica gel column chromatography	GC-ECD	1.0 µg/L (for hexa)	102 (for hexa)	Willet et al. 1978
Whole blood	Extract denatured sample with petroleum ether-ethyl ether; clean up by Florisil column chromatography	GC-ECD	0.7 µg/kg	90–96	Domino et al. 1980
Feces	Extract sample with petroleum ether-ethyl ether; clean up by Florisil and silica gel column chromatography	GC-ECD	1.4 µg/kg (for hexa)	61 (for hexa)	Willet et al. 1978
Bile	Extract denatured sample with petroleum ether-ethyl ether; clean up by Florisil and silica gel column chromatography	GC-ECD	0.08 µg/kg (for hexa)	92 (for hexa)	Willet et al. 1978
Milk	Extract denatured sample with petroleum ether-ethyl ether; clean up by Florisil and silica column chromatography	GC-ECD	1.4 µg/L (for hexa)	108 (for hexa)	Willet et al. 1978
Milk, human	Extract with potassium oxalate, ethanol/diethyl ether, or hexane	GC-ECD	1 µg/kg	No data	Eyster et al. 1983
Liver	Extract sample with methanol-chloroform; clean up by acidic silica column chromatography	GC-ECD	No data	70 (for hexa)	Fawkes et al. 1982
Adipose tissue	Extract sample with methylene chloride; clean up by acidic silica gel column chromatography	GC-ECD	No data	80	Fawkes et al. 1982
Adipose tissue (exposed workers)	Toluene/ethyl acetate (1+3); clean up using GPC/Bio beads	GC-ECD	0.5 µg/kg	98	Wolff et al. 1979

Table 7-1. Analytical Methods for Determining PBBs in Biological Materials *(continued)*

Sample matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Human tissues (post-mortem)	Extract with hexane; clean up using Florisil column	GC-ECD	0.5 ng/g	No data	Micelli et al. 1985

EC = electron capture detection; GC = gas chromatography; GPC = gel permeation chromatography; hexa = hexabrominated biphenyl; PBBs = polybrominated biphenyls; SIM = selected ion monitoring

Table 7-2. Analytical Methods for Determining PBDEs in Biological Materials

Sample matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Animal tissues (muscle, fat, and egg)	Extraction with sulfuric acid; clean up with GPC/silca column/carbon column	GC-MS (NCI)	No data	No data	Sellström et al. 1993
Human adipose tissue	Soxhlet extraction; clean up using 2 solid-phase extraction cartridges	Capillary GC-EILR-MS	0.05–0.30 ng/g lipid	81–103%	Covaci et al. 2002
Human adipose tissue	Extraction with methylene chloride; evaporate; clean up on silica gel followed by clean up on alumina and on a carbon/silica gel column	HRGC/HRMS	0.73–120 ng/kg	No data	Cramer et al. 1990
Human liver/adipose tissue	Extract with 2-propanol/hexane; clean up with Lipidex 5000, column chromatography/GPC	GC-MS (NCI)	5 pg/g lipids	83% (54–116%) liver 71% (51–95%) adipose	Meironyte Guvenius et al. 2001
Human milk	Extract with potassium oxalate/ethanol/diethyl ether/pentane; GPC; clean up on Florisil; elute with hexane	GC/MS (NCI/SIM)	<0.6 ug/kg fat	No data	WHO 1994a
Human milk	Extract by column chromatography using hexane/dichloromethane/ hexane; clean up using GPC	GC-MS (SIM)	5 pg/g lipids	86–102%	Meironyté et al. 1999a, 1999b
Human milk	Extract with n-hexane; clean up using multi-layer column	HRGC-LRMS or LRGC-HRMS (EI-SIM)	No data	>80%	Ohta et al. 2002
Human plasma	Extract with formic acid, 2-propanol, and water on a SPE column; derivatized using diazomethane	GC-MS (NCI)	1–10 pg/g plasma	72%	Thomsen et al. 2001b

Table 7-2. Analytical Methods for Determining PBDEs in Biological Materials *(continued)*

Sample matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Human serum	Extraction with hexane/MTBE (1:1); clean up silica gel/surfuric acid column	GC-ECD; GC-MS (NCI)	0.7 ng/g lipid weight	69–104% (low spike); 77–104% (high spike)	Sjödin et al. 1999a

ECD = electron capture detection; EI = electron input; EILR = electron impact low-resolution; GC = gas chromatography; GPC = gel permeation chromatography; HRGC = high resolution gas chromatography; HRMS = high resolution mass spectrometry; LRGC = low resolution gas chromatography; LRMS = low resolution mass spectrometry; MS = mass spectrometry; MTBE = methyl-tert-butyl either; NCI = negative chemical ionization; PBDEs = polybrominated diphenyl ethers; SIM = secondary ion measurement; SPE = solid phase extraction

specific gas chromatographic detection, or MS (de Kok et al. 1977; Erney 1975; Pomerantz et al. 1978). High recoveries (80–90%) of PBB residues are obtained by the available analytical methods. Typically, the limit of quantitation for PBB residues is about 1 µg/kg in blood serum, 1 µg/kg in human milk, and 0.5 µg/kg in adipose tissue (Eyster et al. 1983; Wolff et al. 1979a). An interlaboratory study is available that validates the precision and accuracy of PBB residue determination in human serum by a commonly used method (Burse et al. 1980).

Polybrominated Diphenyl Ethers. Residues in biological samples can be extracted using sulfuric acid, 2-propanol/hexane, methylene chloride, n-hexane, formic acid/2-propanol/water, or hexane/methyl *t*-butyl ether (Cramer et al. 1990; Meironyte Guvenius et al. 2001; Ohta et al. 2002; Sellström et al. 1993; Sjödin et al. 1999; Thomsen et al. 2001b). Samples are cleaned up to remove interferences using Florosil, silica gel, alumina or activated charcoal column chromatography, gel permeation chromatography (GPC), and/or liquid chromatography (LC) (Sellström et al. 1993; Cramer et al. 1990; Meironyte Guvenius et al. 2001; Sjödin et al. 1999). Most techniques are based on analysis by GC with ECD, or, coupled with MS (WHO 1994a). Capillary columns and temperature programming allow the separation of the different PBDE congeners. High recoveries (69–104%) of PBDE residues are obtained by the available analytical methods. Typically, the limit of quantitation for PBDE residues is about 0.7 ng/g lipid in blood serum, 5 pg/g lipid in human milk, and 0.3 ng/g lipid in adipose tissue (Covaci et al. 2002; Meironyté et al. 1999a, 1999b; Sjödin et al. 1999).

7.2 ENVIRONMENTAL SAMPLES

Most environmental analyses have been performed using multiresidue methods involving solvent extract of the analytes from the sample matrix, clean-up to remove interfering compounds, determination by GC with ECD, and confirmation using an ancillary method such as MS. New methods and technologies are evolving, and this has resulted in lower detection limits. For example, detection limits for PBBs are in the low ppb to ppt range for water matrices and in the low ppm to ppb range for food; for PBDEs, detection limits are in the low ppb range for water matrices and in the low ppb to ppm range for fish tissues. Analytical methods for the determination of PBBs and PBDEs in environmental samples are given in Tables 7-3 and 7-4, respectively.

Polybrominated Biphenyls. Residues in environmental samples can be extracted using hexane-ether, petroleum ether-ether, toluene-ethyl acetate, or methylene chloride (Burse et al. 1980; Domino et al.

Table 7-3. Analytical Methods for Determining PBBs in Environmental Samples

Sample Matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Commercial	Sample dissolved in benzene FireMaster BP-6	GC-ECD	1.6 ng (EC) GC-PED	Not applicable 2.8 ng (PED)	Mulligan et al. 1980
Soil	Extract sample with hexane-acetone; clean up by Florisil column chromatography	GC-ECD	0.1 µg/kg	74.2–83.2 (for hexa)	Jacobs et al. 1976, 1978
Soil	Extraction using hexane/acetone; clean up using Florisil column	GC-FID/ECD	No data	No data	Hill et al. 1982
Plant tissue	Extract macerated sample with hexane-acetone; clean up by Florisil column chromatography	GC-ECD	0.3 µg/kg	No data	Jacobs et al. 1978
Effluent and river water	Extract sample with hexane-ethyl ether	GC-ECD	0.1 µg/kg	90	Hesse and Powers 1978
Sediment	Extract sample with hexane-acetone	GC-ECD	No data	No data	Hesse and Powers 1978
Sediment	Pressurized hot water extraction coupled with clean up by LC	LC-GC-MS/FID	0.71 ng/g	No data	Kuosmanen et al. 2002
Fish	Extract homogenized sample with hexane-water; clean up by acidic and basic silica columns	GC-ECD	No data	98 (for hexa)	Gobas et al. 1989
Fish	Extract homogenized sample with hexane-methylene chloride; clean up by gel permeation and silica gel chromatography	HRGC-HRMS	No data	No data	Kuehl et al. 1991
Fish	Extract homogenized sample with hexane-acetone; clean up by gel permeation chromatography	HRGC-MS/NCI and HRGC-ECD	No data	No data	Jaffe et al. 1985
Terrestrial, fresh water, and marine samples	Extraction with diethyl ether/hexane; hydrolysis with 98% sulfuric acid/bio beads/silica gel/activated charcoal	MS (NCI)	No data	No data	Jansson et al. 1991, 1993
Dolphin fat	Soxhlet extraction using hexane-methylene chloride; clean up using GPC, silica gel	MS	No data	No data	Kuehl et al. 1991
Animal feeds	Elute ground sample containing celite with methylene chloride; clean up by Florisil column chromatography	GC-ECD	8 µg/kg (for hexa)	98 (for hexa)	Fehringer 1975b
Dairy products	Fat extracted by methanol/ether; clean up by GPC, 25% toluene in ethyl acetate	GC-ECD	7 µg/kg	No data	Fehringer 1975

Table 7-3. Analytical Methods for Determining PBBs in Environmental Samples *(continued)*

Sample Matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Plants	Cut, extracted with hexane/acetone; clean up with Florisil column	GC-ECD	0.3 µg/kg wet basis	No data	Chou et al. 1978

EC = electron capture detection; FID = flame ionization detector; GC = gas chromatography; hexa = hexabrominated biphenyl; HRGC = high resolution gas chromatography; HRMS = high resolution mass spectrometry; LC = liquid chromatography; MS = mass spectrometry; NCI = negative chemical ionization; PED = plasma emission detection; PBBs = polybrominated biphenyls

Table 7-4. Analytical Methods for Determining PBDEs in Environmental Samples

Sample Matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Air	Air pumped through glass fiber filter and adsorbent trap; filters and adsorbants are Soxhlet extracted with acetone/hexane; cleaned-up by column chromatography	GC/MS	No data	No data	Dodder et al. 2000a
Water	Clean up by disk-type C18 solid-phase extraction	Capillary GC-ECD	0.12 ng/mL	103±8.6% (river water) 87±10.7% (sea water)	Yamamoto et al. 1997
Sewage	Extract with chloroform; evaporate and dissolve residue in ethanol	GC/MS	0.06 mg/kg	No data	WHO 1994a
Sediment	Clean up by cartridge-type Florosil extraction	Capillary GC-ECD	9.7 ng/g	91±6.3%	Yamamoto et al. 1997
Sediment	Pressurized hot water extraction coupled with clean up by LC	LC-GC-MS/FID	0.71 ng/g	No data	Kuosmanen et al. 2002
Sediment	Extract with acetone; clean up on Florisil	NAA GC/EC	<5 µg/kg <5 µg/kg	No data	Watanabe et al. 1987
Fish	Extract with acetone-hexane + hexane-ethyl ether; treatment with sulfuric acid or clean up on alumina; chromatography on silica gel	GC/EC; GC/MS	0.1 mg/kg fat	No data	Anderson and Blomkvist 1981
Fish	Extract with dichloromethane on chromatography column; clean-up using GPC; fractionation using silica gel column	GC-HRMS (NCI)	5–93 pg/g	No data	Alaee et al. 2001
Fish	Extract clean up with GPC and mini-column chromatography; concentration	GC-MS (NCI)	0.01–0.2 ng/g lipid	88–128%	Akutsu et al. 2001

Table 7-4. Analytical Methods for Determining PBDEs in Environmental Samples

Sample Matrix	Preparation method	Analytical method	Sample detection limit	Percent recovery	Reference
Animal tissues	Homogenize; extract with n-hexane-acetone; treatment with sulfuric acid; GPC; chromatography or silica gel chromatography or activated charcoal	GC/MS (NCI)	10 ng/kg	No data	Jansson et al. 1991

ECD = electron capture detection; GC = gas chromatography; GPC = gel permeation chromatography; HRMS = high resolution mass spectrometry; LC = liquid chromatography; MS = mass spectrometry; NAA = neutron activation analysis; NCI = negative chemical ionization; PBDEs = polybrominated diphenyl ethers

1980; Wolff et al. 1979b; Fawkes et al. 1982; Fehringer 1975b). As for biological samples, quantitation of environmental samples is also usually done by GC. Capillary columns are required for the separation of the individual congeners in a mixture (Robertson et al. 1983b). High recoveries (74–98%) of PBB residues in environmental samples are obtained by the available analytical methods. Typically, the limit of quantitation for PBB residues is about 0.1 µg/kg in soil and 0.7 µg/kg in sediment (Jacobs et al. 1976, 1978; Kuosmanen et al. 2002).

Polybrominated Diphenyl Ethers. Like PCBs, air samples containing PBDEs are usually collected by pumping air through a sampler containing a glass fiber filter and adsorbent trap to separate the particle bound and vapor phase fractions, respectively (Dobber et al 2000a; Hillery et al 1997). The filters and adsorbants are then Soxhlet extracted with acetone/hexane, and the extracts are cleaned-up and analyzed by high resolution GC techniques.

Residues in environmental samples can be extracted using chloroform, acetone, acetone-hexane, hexane-acetone, and hexane-ether (Anderson and Blomkvist 1981; Jansson et al. 1991; Watanabe et al. 1987; WHO 1994a). Samples are cleaned up to remove interferences using Florosil, silica gel, alumina or activated charcoal column chromatography, GPC, and/or LC (Akutsu et al. 2001; Alaee et al. 2001; Anderson and Blomkvist 1981; Jansson et al. 1991; Watanabe et al. 1987; Yamamoto et al. 1997). As for biological samples, quantitation of environmental samples is also usually done by GC. Capillary columns are required for the separation of the individual congeners in a mixture (WHO 1994a). High recoveries (88–128%) of PBDE residues in environmental samples are obtained by the available analytical methods (Akutsu et al. 2001). Typically, the limit of quantitation for PBDE residues is about 0.12 ng/mL in water, 9.7 µg/kg in sediment, and 0.2 µg/kg lipid in fish (Akutsu et al. 2001; Yamamoto et al. 1997). The first interlaboratory study on PBDEs in environmental samples showed that there is good agreement for quantification of BDE-47 and BDE-100 congeners. However, improved methods are required for analysis of BDE-99, -153, -154, and -209 congeners (de Boer 2000).

7.3 ADEQUACY OF THE DATABASE

Section 104(i)(5) of CERCLA, as amended, directs the Administrator of ATSDR (in consultation with the Administrator of EPA and agencies and programs of the Public Health Service) to assess whether adequate information on the health effects of PBBs and PBDEs is available. Where adequate information is not available, ATSDR, in conjunction with the National Toxicology Program (NTP), is required to

assure the initiation of a program of research designed to determine the health effects (and techniques for developing methods to determine such health effects) of PBBs and PBDEs.

The following categories of possible data needs have been identified by a joint team of scientists from ATSDR, NTP, and EPA. They are defined as substance-specific informational needs that if met would reduce the uncertainties of human health assessment. This definition should not be interpreted to mean that all data needs discussed in this section must be filled. In the future, the identified data needs will be evaluated and prioritized, and a substance-specific research agenda will be proposed.

7.3.1 Identification of Data Needs

Methods for Determining Biomarkers of Exposure and Effect.

Exposure. Methods used as biomarkers for exposure to PBBs and PBDEs are available (Brilliant et al. 1978; Covaci et al. 2002; Eyster et al. 1983; Landrigan et al. 1979; Meironyté et al. 1999a, 1999b; Sjödin et al. 1999; Wolff et al. 1982). Analytical methods of sufficient precision and accuracy are presently available for the determination of PBBs and PBDEs in adipose tissue, serum, and breast milk (Burse et al. 1980; Covaci et al. 2002; Domino et al. 1980; Fawkes et al. 1982; Fehringer 1975a; Meironyté et al. 1999a, 1999b; Sjödin et al. 1999; Willet et al. 1978; Wolff et al. 1979a, 1979b). Addition congener standards are needed for PBB and PBDEs analysis. Only 30–40 congener standards are currently available for identification and quantification of PBDEs (Eljarrat et al. 2002; Sjödin et al. 1998). Metabolites are also important biomarkers for exposure to PBBs and PBDEs. However, these compounds are mostly unknown, and standards are not available.

Effect. No known effect of PBB or PBDE exposure can be quantitatively correlated with PBB or PBDE exposure.

Methods for Determining Parent Compounds and Degradation Products in Environmental Media. Analytical methods of sufficient sensitivity are presently available for the determination of PBBs and PBDEs in environmental samples (Akutsu et al. 2001; Anderson and Blomkvist 1981; Fehringer 1975b; Hesse and Powers 1978; Jacobs et al. 1976, 1978; Yamamoto et al. 1997).

It would be helpful to develop data determining the detection limit and accuracy of PBB determinations in fish and other aquatic animals (e.g., seals) and in sediment (Gobas et al. 1989; Jaffe et al. 1985; Kuehl

et al. 1991). Analytical methods for determining lower brominated PBBs in environmental samples are available (Morris et al. 1992). An analytical method to determine PBB metabolites in fish would be helpful. A method for determining of 6-hydroxy-2,2',4,4',5,5'-hexabromobiphenyl, a metabolite of 2,2',4,4',5,5'-hexabromobiphenyl, in dog feces is available (Gardner et al. 1979). Photochemical degradation leads to the formation of lower brominated products, which are the only environmental degradation products identified for PBBs. Analytical methods are presently available for the determination of these compounds in environmental samples (De Kok et al. 1977; Hill et al. 1982; Robertson et al. 1983b). There is no evidence in the literature of detectable biodegradation of PBBs in the environment under aerobic conditions (Griffin and Chou 1981a, 1981b), but the compounds may biodegrade to debrominated products under anaerobic conditions in polluted environments (Morris et al. 1992).

It would be helpful to develop data determining the accuracy of PBDE determinations (e.g., percent recovery) in environmental samples. Methods for determining degradation products and metabolites of PBDE are needed. There is no information in the literature of detectable biodegradation of PBDEs in the environment under aerobic or anaerobic conditions. The analysis of PBDE pyrolysis degradation products, such as polybrominated dibenzo-*p*-dioxins and dibenzofurans (PBDD/Fs), is often disturbed by the presence of PBDEs. Ebert et al. (1999) demonstrated that by using a Florisil column in a sample clean-up process, almost complete separation of PBDEs and PBDD/Fs is achieved before analysis by GC-MS.

7.3.2 Ongoing Studies

Two ongoing studies regarding analytical methods for determining PBB and PBDE residues or metabolites were located. This information is presented in Table 7-5, and was found as a result of a search of the Federal Research in Progress Database (FEDRIP 2002).

Table 7-5. Ongoing Studies on Analytical Methods for PBBs and PBDEs

Investigator	Affiliation	Subject	Sponsor
Huwe JK et al.	Agricultural Research Service, Fargo, North Dakota	Dioxins and other environmental contaminants in foods	USDA
Robertson LW	University of Kentucky Medical Center	Synthesis	National Institute of Environmental Health Sciences

Source: FEDRIP 2002

PBBs = polybrominated biphenyls; PBDEs = polybrominated diphenyl ethers; USDA = U.S. Department of Agriculture